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Facile Synthesis of 3D Porous SnO_2 Nano Flakes by a Simple Hydrothermal Method and Their Ethanol Gas Sensor Properties

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***Abstract*— 3D porous flake-like SnO_2 was successfully synthesized through a very simple and low-cost hydrothermal method followed by calcination process and characterized by Scanning Electron Microscopy (SEM), energy dispersive X-ray (EDX), Transition Electron Microscopy (TEM), High Resolution Transition Electron Microscopy (HRTEM), and X-ray diffraction (XRD) instruments. Moreover, the sensor was evaluated to test in various ethanol concentrations and operating temperatures. The sensor based on these (3D) porous SnO_2 nanoflakes exhibited good sensitivity and selectivity toward ethanol gas at operating temperature 200 °C. The response time and recovery time of the sensor were about 6 s and 5 s to 500 ppm ethanol, respectively. These findings indicated that (3D) porous SnO_2 nanoflakes could be used as a candidate to fabricate ethanol sensors in practical applications.**

Keywords---**ethanol, hydrothermal, nanoflakes, sensor, SnO_2.**

I. INTRODUCTION

In recent years, metal-oxide-semiconductors such as ZnO, SnO_2, TiO_2, etc., have attracted great deal of interest in gas-sensing research field on account of their low cost, and high response to the target gasses [1]-[4]. Among them, tin oxide SnO_2 is regarded as a very important oxide material with a wide n- type band gap of ≈3.6 eV, at 300K, it has been demonstrated as one of most promising metal oxides for the fabrication of gas sensor with high sensitivity, excellent stability and low price [5],[6]. There have been tremendous reports on the morphology, microstructure and electronic properties of the gas-sensing materials due to their role in the material sensitivity, selectivity and stability which were considered as key factors of the gas sensing performance [7]–[9]. Constructing the (3D) porous SnO_2 nanoflakes aims to increase interactive surface and provide more active sites which leads to achieve high and quick gas response. Moreover, surfactants have also been used to improve the sensor performance by controlling materials morphology [10]. SnO_2 sensors have been used extensively in gas detection field. However, they are usually used at high operating temperatures (above 300°C). Thus, it is worth to think about producing SnO_2 sensors operating at relatively low temperatures.

Herein, (3D) Porous flake-like SnO_2 with excellent ethanol sensing properties at operating temperature of (200°C) are synthesized hydrothermally using CTAB as a surfactant followed by calcination process. Moreover, the structural, morphological and sensing properties of the as-synthesized nanoflakes have been investigated.

II. EXPARIMENTAL

In a typical procedure, 1.231g $SnCl_2$ $.2H_2O$ was first dissolved under stirring into 30 mL deionized water. Next, 1.808g CTAB and 0.599g NaOH were added into above solution under stirring at 25°C for 30 min. Then, The hydrothermal growth was carried out at 130 °C for 22 h in a hydrothermal synthesis reactor, after hydrothermal process, the autoclave cooled down to room temperature naturally. The resulting product was washed with distilled water and absolute ethanol several times and then dried in vacuum at 60°C for 11 h, followed by calcination process in a furnace at 600°C for 3 h at a ramping rate of 10°C/min. Finally, SnO_2 powders were obtained.

Crystal structure of (3D) porous SnO_2 nanoflakes were determined by X-ray diffraction (XRD) using an X-ray diffractometer (XRD, D/Max-2400) with Cu Kα1 radiation ($\lambda = 0.15406$ nm). Elemental composition was examined by energy-dispersive X-ray detector (EDX). Morphological analysis was carried out on a scanning electron microscopy (SEM, S-4800) and transmission electron microscopy (TEM, JEM-2010). The gas sensing properties were evaluated by the WS-30B gas sensing apparatus (Wei Sheng Electronics Science and Technology Co., Ltd., Henan Province, China). The sensor response (R) to gas was defined as Ra/Rg, where Ra and Rg were the initial sensor resistance in air and gas, respectively [1].

III. RESULTS AND DISCUSSION

The X-ray diffraction (XRD) analysis is performed to investigate the crystal structure of our sample. Fig. 1(a) Shows XRD patterns of (3D) porous SnO_2 nanoflakes. The peak positions of the sample exhibit the rutile type tetragonal structure of SnO_2, which were indexed with standard card (JCPDS, 41-1445) with $a = b = 4.736$ Å and $c = 3.185$ Å. The strong diffraction intensity indicates high crystallinity of the sample after calcining at 600°C for 3 h. No impurity phase detected which indicates the high purity of the prepared SnO_2 [11]. Fig. 1(b) illustrates the EDX spectroscopy of (3D)

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porous SnO2 nanoflakes indicating that it is composed of Sn and O elements (the presence of signals of Au can be ascribed to the Au grid). Fig. 2 (a) and (b) represents the SEM and high magnification SEM images of the as-synthesized product, respectively. It can be readily seen that the sample is flake-like in porous texture. Fig. 2(c) displays the TEM image of the as- prepared sample. It can be clearly seen that the sample is composed of thin nanoflakes, which is corresponding to the results of SEM. Fig. 2(d) is the HRTEM image, which clearly shows that the lattice distance is 0.336 nm. It corresponds to (110) crystallographic orientation.

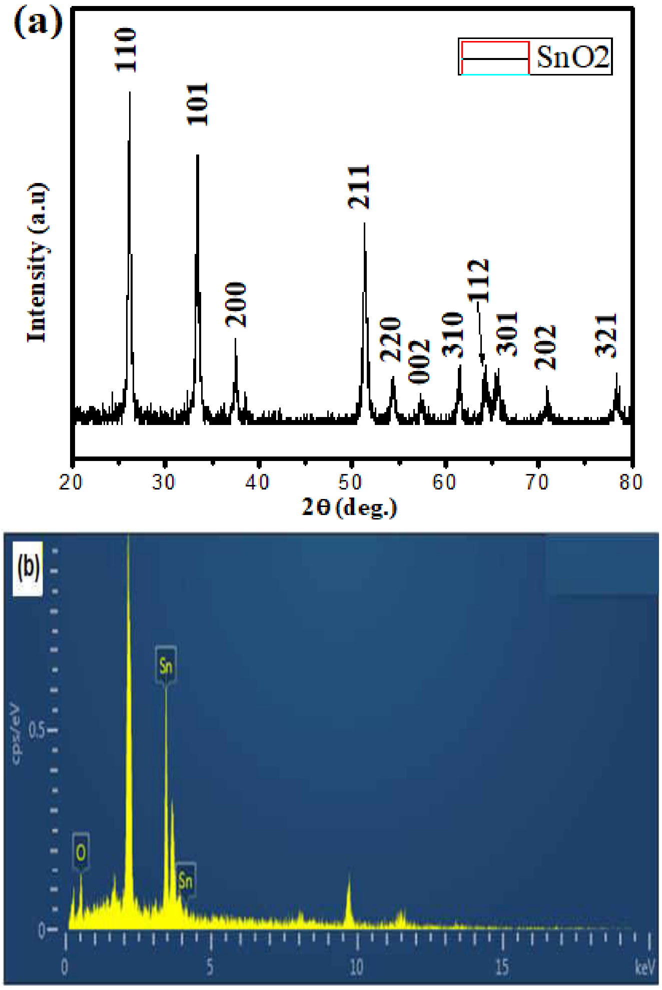
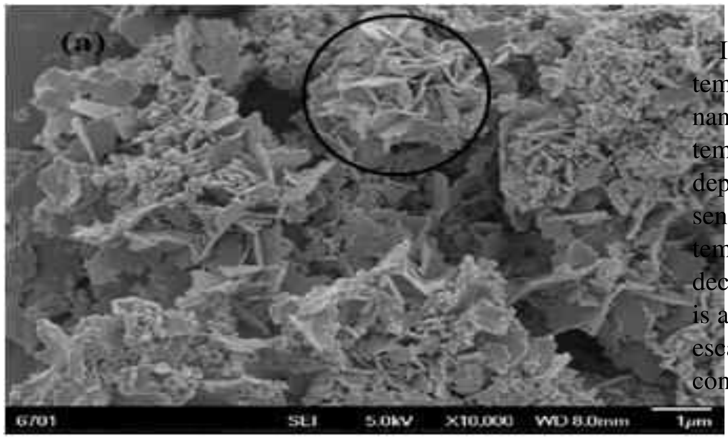


Fig.1. (a) XRD and (b) EDS patterns of (3D) porous SnO2 nanoflakes.



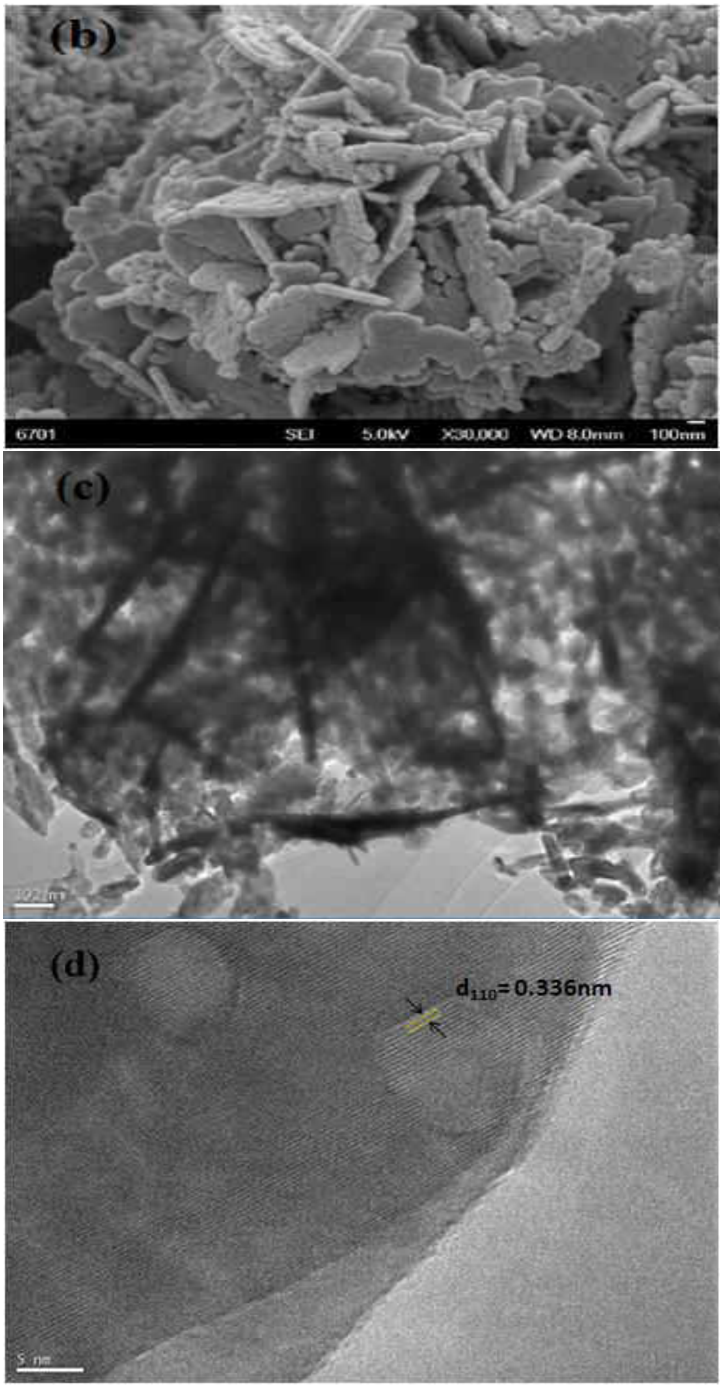
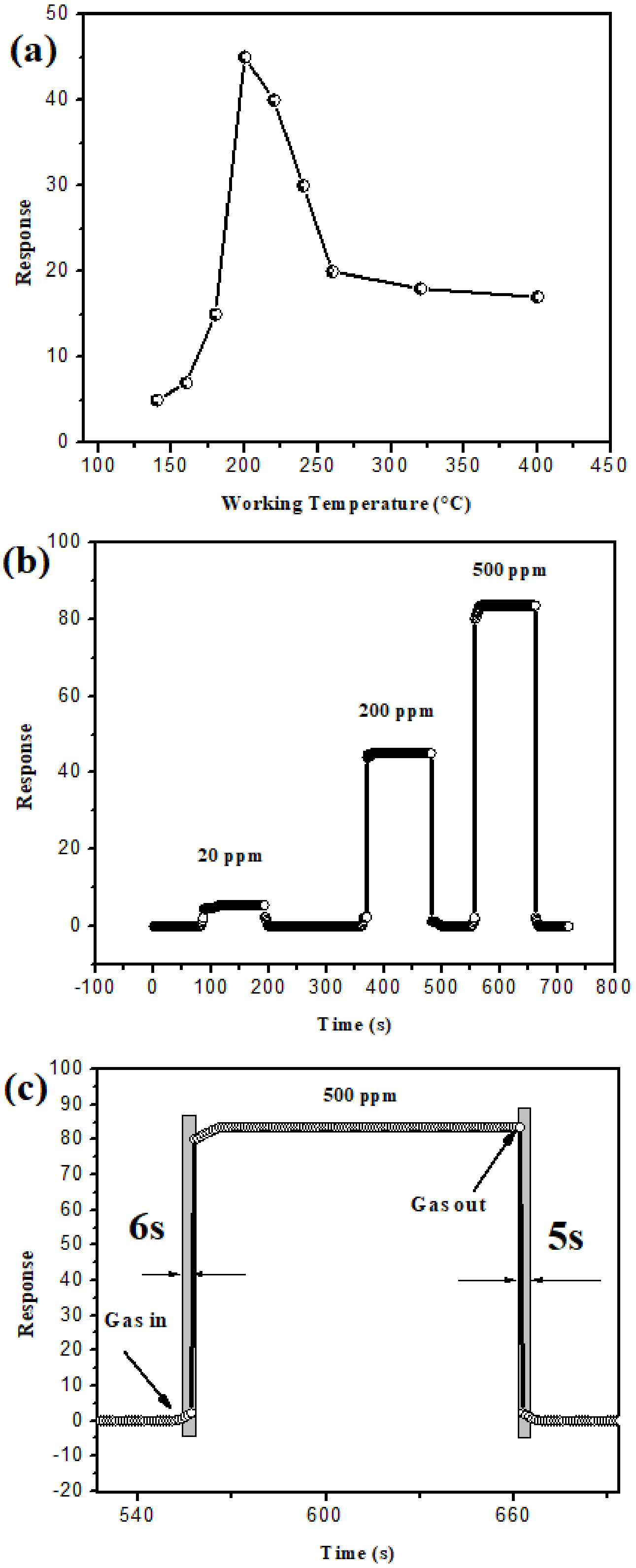


Fig.2. (a) SEM and (b) high magnification of SEM; (c) TEM image and (d) HRTEM of (3D) porous SnO_2 nanoflakes.

In order to determine the sensors optimum operating temperature, the response values of (3D) porous SnO_2 nanoflakes to 200 ppm ethanol under different operating temperatures in the range of 140–400 °C are evaluated and depicted in Fig. 3(a). As indicated in the figure, from 160°C, sensor response rapidly increases as the operating temperature increases. Reaching its peak at 200 °C it starts to decrease. The reason of poor response when the temperature is above 200 °C is that number of adsorbed oxygen ions will escape before reactions take place, leading to increase conduction electron density [12].



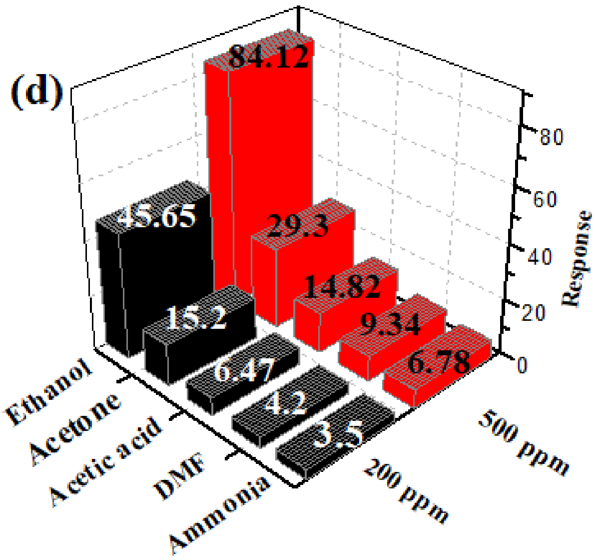


Fig. 3. (a) The response to 200 ppm ethanol at different operating temperatures (200-400 °C), (b) the response of (3D) porous SnO_2 nanoflakes to 20, 200 and 500 ppm ethanol at 200 °C, (c) dynamic sensing transient of the sensor to 500 ppm for ethanol and (d) sensor responses to 200 and 500 ppm different gases at 200 °C.

Fig. 3(b) displays the dynamic response/recovery plots of (3D) porous SnO_2 nanoflakes under different gas concentrations from 20 to 500 ppm at 200 °C for ethanol. It can be clearly seen that the response increases with increasing concentration of ethanol. Moreover, it is worth to note that our sensor can show a considerable response (about 5.4 for ethanol) while the gas concentration is low (20 ppm). This result proves that the gas-sensing property of the as-prepared (3D) porous SnO_2 nanoflakes is good. The transient characteristics of the (3D) porous SnO_2 nanoflakes sensor in the ethanol atmosphere at 500 ppm are shown in Fig. 3(c). The response and recovery times are about 6 s and 5 s, respectively. And this result is better compared with some previous reports on the sensing properties [13]-[15]. Furthermore, the selectivity is also an important parameter to assess the properties of sensors. Fig. 3(d) shows the responses of (3D) porous SnO_2 nanoflakes to 200 and 500 ppm different gasses at 200 °C. Our sensor exhibits high selectivity to ethanol. The results indicate that the (3D) porous SnO_2 nanoflakes based sensor can successfully distinguish ethanol at 200 °C.

IV. CONCLUSION

In summary, (3D) porous SnO_2 nanoflakes have been successfully synthesized through a facile and low-cost hydrothermal method and followed by calcination. The sensor exhibits excellent ethanol sensing performances due to the porous flake-like nanostructures. We have also systematically investigated the ethanol sensing properties of this sample. The results show that our product found to be composed of (3D) porous nanoflakes, which consequently results in the fast response/recovery time and good sensitivity and excellent selectivity to ethanol at 200 °C. Thus (3D) porous SnO2 nanoflakes can be used as a promising material for ethanol sensors.

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